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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Shouquan Huo, et al

ORGANIC ELEMENT FOR
ELECTROLUMINESCENT DEVICES

Serial No. 10/729,238

Filed 05 December 2003

Group Art Unit: 1774

Examiner: Dawn L. Garrett

I hereby certify that this correspondence is being deposited today with the United States Postal Service as first class mail in an envelope addressed to Commissioner For Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Deidra L. Mack
Deidra L. Mack

June 14, 2005
Date

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION PURSUANT TO 37 C.F.R. 1.131

The undersigned, Shouquan Huo, states that:

he is a co-inventor of the claimed subject matter of the above-referenced patent application, hereinafter referred to as "the invention";

he has read and is familiar with the cited reference, Williams et al., INORGANIC CHEMISTRY, (2003), Vol. 42, pages 8609-8611, and, according to the first page of the article, it was "Published on the Web" on 11/20/2003;

on or prior to November 20, 2003, and at the time the invention occurred, he was an employee of the Eastman Kodak Company in Rochester, New York, assigned to conduct research in the area of light emitting OLED devices.

on or prior to November 20, 2003, the undersigned and his coinventors conceived and actually reduced to practice the invention at the above mentioned Kodak facility in the United States;

this is demonstrated by the submission of contemporaneous records dated on or before November 20, 2003 relating to the preparation of compounds and evaluation of those compounds as light emitting compounds including the following:

pages 4, 19, 27, and 29 describes the making of the compound corresponding to Inv-1 (same as L¹ in Williams);

pages 32 describes an OLED device prepared using compound Inv-1 and shows the high luminance efficiency obtained with emitter Inv-1;

pages 43 and 44 describe the preparation of variants of Inv-1 with different anionic groups and transition metals;

each of these pages was witnessed as read and understood on or prior to November 20, 2003; and

it is believed that compounds L^2 and L^3 are obvious variants of L^1 and within the generic invention suggested by the discovery of Inv-1.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 6/14/05



Shouquan Huo

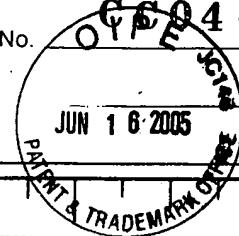
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Notebook No.

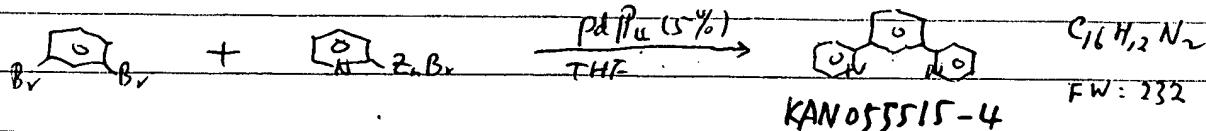
Date

Problem:



RESEARCH / DEVELOPMENT

EASTMAN KODAK COMPANY



In a three-necked flask (dried under N_2) were charged THF (10 mL), 1,3-dibromobenzene (1.4 mL, 10 mmol), and $\text{Pd}(\text{PPh}_3)_4$ (0.51 g, 0.44 mmol) under N_2 . To the mixture was added 48 mL (0.5 M, 24 mmol) of 2-pyridylzinc bromide via a syringe at room temperature. The resulting ^{then at -70°C} dark-brown was stirred at room temperature overnight. After usual work up and acidic ^{basic} extraction. The crude product was purified by column on silica gel using $\text{CH}_2\text{Cl}_2 : \text{EtOAc} = 8:1$. 1.78 g, 76.7%.

KP 15226-6/00

Signature

Shoung Hsu

The foregoing disclosed to me or _____

Witness Ronald E. Leone

EASTMAN KODAK COMPANY

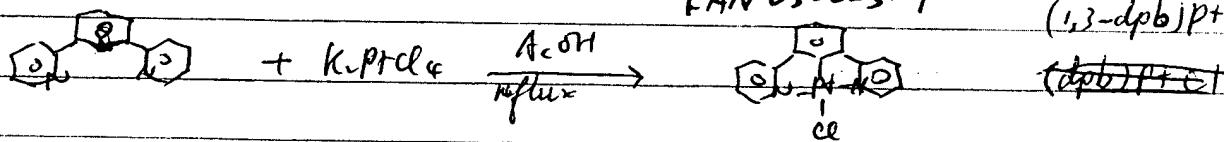
Notebook No. _____

Date _____

Problem: Preparation of tridentate Pt complex.

A mixture of 0.38g (1.64 mmol) of 1,3-dipyridylbenzene, 0.67g (1.62 mmol) of KPtCl_4 , and 400 ml of AcOH was degassed and then refluxed (110-120°C bath) for 4:00 pm - 2 days. The reaction mixture was cooled to rt, the precipitates were collected by filtration and washed with MeOH, water, and EtOH, and CH_2Cl_2 and dried in air. 0.58g.

KAN 055225-7

 $(1,3\text{-dpb})\text{PtCl}_2$ 

The material was extracted with 100 ml of CH_2Cl_2 . The extract was introduced on a prepared column and eluted with $\text{CH}_2\text{Cl}_2 : \text{EtOH} = 9:1$ (v/v) (flash). The yellow-orange band was collected and evaporated to give yellow solid, 0.38g. 51%, did not melt at 265-270°C.

The compound was successfully sublimed at 260-270°C. (see page 27)

KP 15226-6/00

Signature _____

Shoung Huo

The foregoing disclosed to me on _____

Witness _____

Ronald E. Leone

RESEARCH / DEVELOPMENT

EASTMAN KODAK COMPANY

Notebook No. CC0465

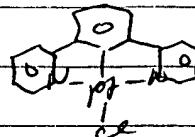
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Date _____

Problem: Sublimation of $\text{pt}(\text{dpb})\text{Cl}$ (CC0465-19)

0.3 g of the compound

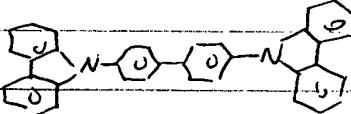


was sublimed at

250°C - 260°C, 4 h, 270°C, 2 h. 0.22 g of yellow crystals were obtained.

9/27/03:

Sublimation of first material CBP



2.07 g. at 265°C. overnight

4,4'-N,N'-dicarbazole-biphenyl.

KP 15226-6/00

Signature Shouquan Han

The foregoing disclosed to me on _____

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RESEARCH / DEVELOPMENT

Notebook No.

CC0465

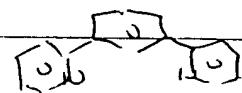
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EMAN KODAK COMPANY

Date

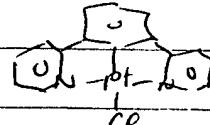
Problem:



2.44g

+ K_3PbCl_6

4.12g

 $\xrightarrow{\text{AcOH (300 mL)}}$
reflux 3 days $(1,3\text{-dipb})\text{pt+Cl}$

0.85 g crude

KP 15226-6/00

Signature

Shouqian Kao

The foregoing disclosed to me on

Witness

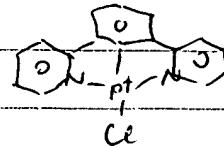
Ronald E. Leone

CC0465. RESEARCH / DEVELOPMENT

EASTMAN KODAK COMPANY

Date _____

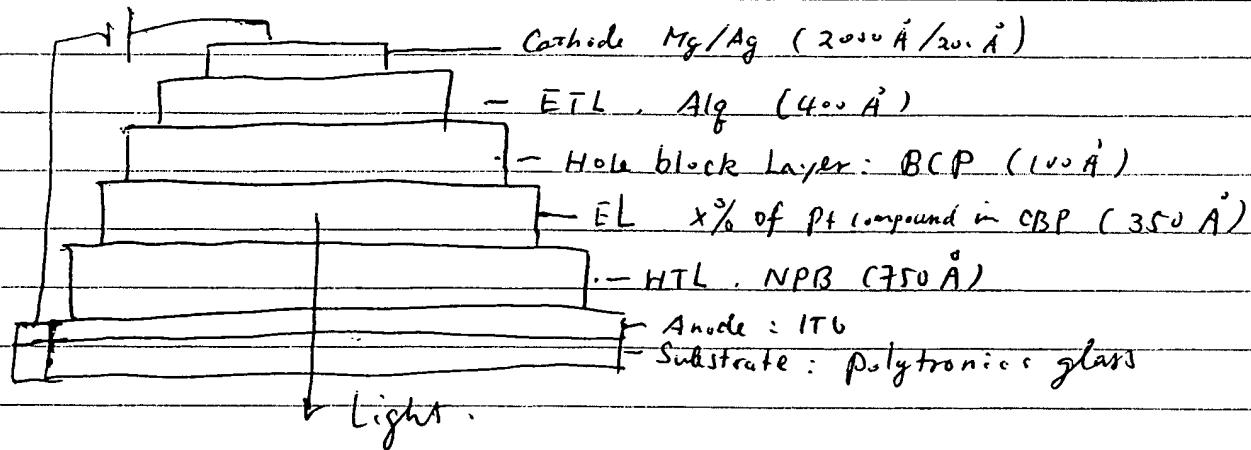
Problem: Test (1,3-diphenyl-1,2,4,5-tetraphenylphosphine) as phosphorescent dopant in CBP



KAN: 055225-7

NB: CC0465-19

Ce



10/22/03

dopant level yield (cd/A) at 20 mA/cm²

Device #	SH031022-1A1	0	0.57
	SH031022-1B1	2%	16.01
	SH031022-1C1	4%	17.55 (19.47 at 2 mA/cm ²)
	SH031022-1D1	6%	15.08
	SH031022-1E1	8%	11.59
	SH031022-1F1	10%	10.62

KP 15226-6/00

Signature

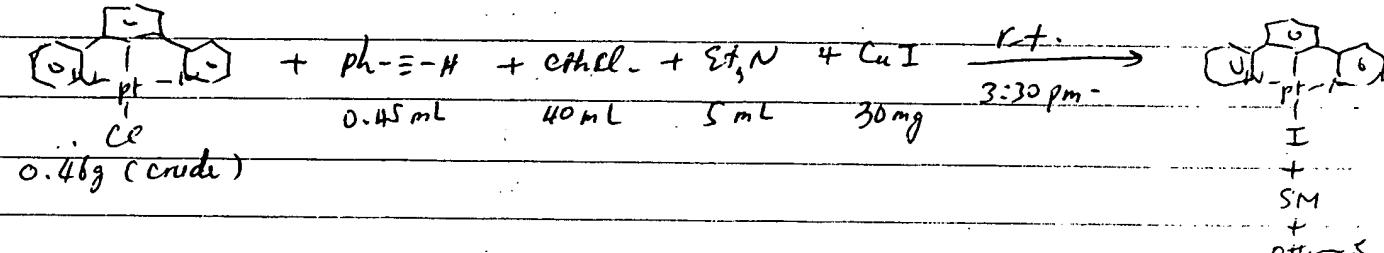
Shing Hsu

The foregoing disclosed to me on

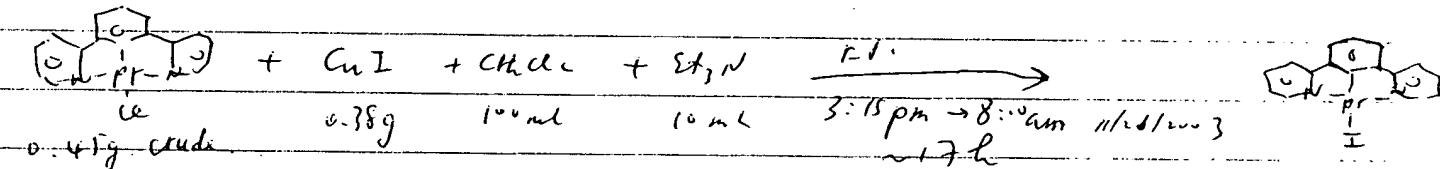
Witness Ronald E. Leone

Problem: Preparation of (ACN)PtI and device test

Date _____



All materials were combined in a flask. The mixture was stirred at room temperature under N_2 in the absence of light (protected from light with aluminum foil). 3:30 pm - overnight. Starting material still remained. 20 mg CuI was added and stirring was r.t overnight. SM still remained. Column separation gave 0.12g of LptI and 0.15g of Lpt-Cl. In another run, 0.2g of Lpt-Cl was stirred with 0.2g of CuI in CHCl₃-Et₃N (50-55 mL) for 24 h, nearly complete I-Cl exchange took place.



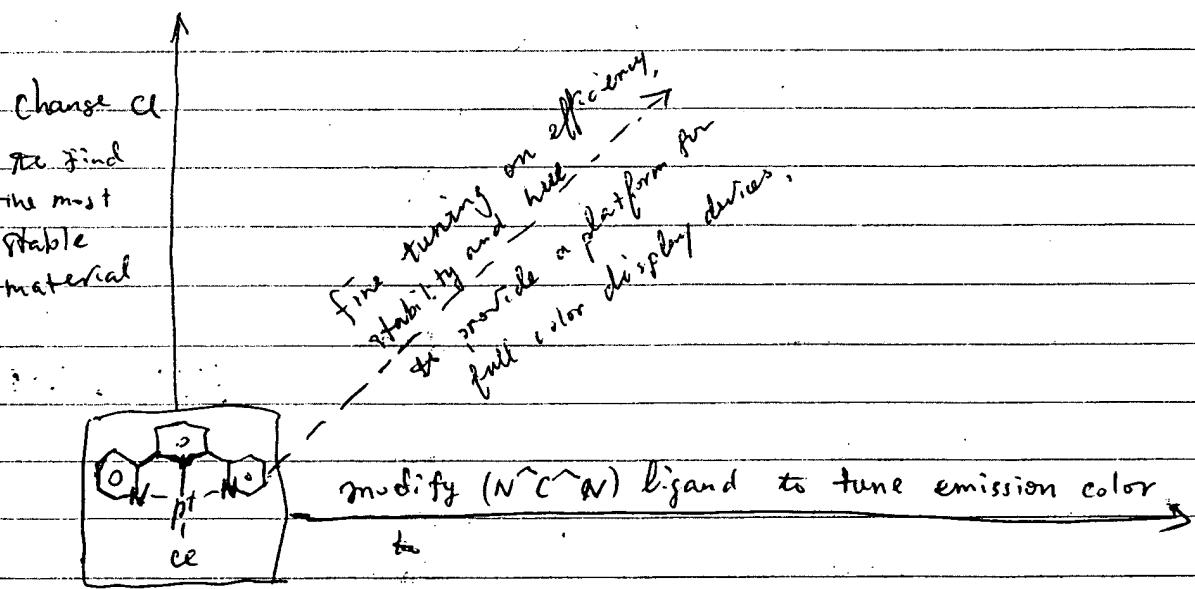
KAN05-6225-9

The mixture was transferred into a one-necked flask. Solvents were removed by rotary evaporation and the residue was treated with EtCl. Flash chromatography on silica gel with EtCl as eluent first to collect Pt-I, then with CHCl₃: EtOAc = 10:1 as eluent to collect Pt-Cl (starting material) [Note: the reaction should be kept for longer time for completion]. 0.15g of starting material was recovered. Pt-I was combined with those from the run before, 0.35g crude. Sublimed at 250-260°C give 0.25g pure sample for NMR, UV, IR, and device fabrication.

12/9/03 Submitted for device fabrication. Host: CBP, Hole blocker: BCP; Hole transport: NPB; ETL: Al₂.

Problem: Design of new tridentate ligands for Pt, Ir, and other transition metals.

Tridentate ($N^{\wedge}C^{\wedge}N$) platinum complexes have been demonstrated as highly efficient triplet emitters (see page 32) and a related class of tridentate ($N^{\wedge}N^{\wedge}C$) platinum acetylidide complexes were also shown valuable potential for OLED applications (see: Wei Lu et al., Chem. Comm. 2002, 206-207). However, although the efficiency was high (17.55 cd/A at 20 mA/cm² and 19.09 at 20 mA/cm² at 4% current level) in the case of ($N^{\wedge}C^{\wedge}N$)PtCl, the stability of the device was rather poor (ca. 1 h half life at 20 mA/cm² current density), an extensive research needs to be carried out to tackle this problem. Based on the current available results, the research can be 3D (three-dimensional) as shown below.



On the other hand, the design of new type of tridentate ligand is still one of our priority in triplet emitter research. One variation on ($N^{\wedge}C^{\wedge}N$) and ($N^{\wedge}N^{\wedge}C$) ligands is the use of P instead of N as donor atom. Either one N or both of N in the ligands can be replaced by P. Some prototype ligands are shown below: (next page)

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